



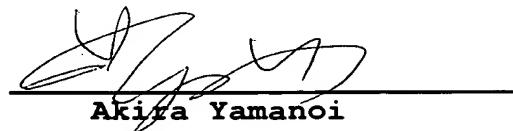
DECLARATION

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do hereby solemnly and sincerely declare:-

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Signed this 17th day of June, 2005.



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[Title of Document] Specification

[Title of the Invention] AQUEOUS INK COMPOSITION

[Scope of Claim for a Patent]

[Claim 1] An aqueous ink composition, characterized  
5 by containing a quick-drying property imparting agent  
which accelerates drying of the ink.

[Claim 2] An aqueous ink composition according to  
claim 1, wherein melting point of the quick-drying  
property imparting agent is in the range of 20-250°C.

10 [Claim 3] An aqueous ink composition according to  
claim 1, wherein the quick-drying property imparting  
agent is a benzotriazole compound having a melting point  
of 20-250°C.

[Claim 4] An aqueous ink composition according to  
15 claim 1, wherein it contains water and a water-soluble  
solvent other than water and the quick-drying property  
imparting agent has a solubility in the water lower than  
a solubility in the water-soluble solvent other than  
water.

20 [Claim 5] An aqueous ink composition according to  
claim 1, wherein it contains water and a water-soluble  
solvent other than water and solubility of the quick-  
drying property imparting agent in water is in the range  
of 0.1-60 wt%, and solubility of the quick-drying  
25 property imparting agent in the water-soluble solvent

other than water is in the range of 0.5-80 wt%.

[Claim 6] An aqueous ink composition according to claim 1, wherein content of the quick-drying property imparting agent in the ink composition is in the range 5 of 1-10 wt%.

[Claim 7] An aqueous ink composition according to claim 1, wherein the water-soluble solvent is at least one solvent selected from the group consisting of alcohol, ketone and ether solvents.

10 [Claim 8] An aqueous ink composition according to claim 1, wherein boiling point of the water-soluble solvent is not higher than 100°C.

[Claim 9] An aqueous ink composition according to claim 1, wherein water content in the composition is 30-15 95%.

[Claim 10] An aqueous ink composition according to claim 1, which additionally contains a fluorescent dye and this dye contains rare earth element.

20 [Claim 11] An aqueous ink composition according to claim 1 which contains a resin and this resin is at least one resin selected from the group consisting of polyvinylpyrrolidone, polyvinyl alcohol, polyurethane, polyacrylic acid, polyether and copolymers thereof.

[Detailed Description of the Invention]

25 [0001]

[Technical Field Pertinent to the Invention]

The present invention relates to an aqueous ink composition which is excellent in drying property and mild for earth environment. More particularly, it relates to an aqueous fluorescent ink composition which 5 is excellent in quick-drying property and mild for earth environment.

[0002]

[Prior Art]

Recently, developments have been extensively 10 made utilizing properties of fluorescent materials, such as various cards used for security, factory automation, for material handling, and the like. One example is utilization for mailing, namely, bar codes are printed with an ink containing a fluorescent material, and 15 postal matters are sorted by code control and distributed.

[0003]

As the fluorescent materials, there have been used fluorescent complexes comprising a rare earth 20 element as a luminescence center with which a low molecular ligand is coordinated, and inks containing them are prepared. These are disclosed, for example, in JP-B-54-22336, JP-A-9-188835, and JP-A-11-510213. These fluorescent complexes are homogeneously dissolved as 25 dyes in liquids such as water and solvents to prepare inks.

[0004]

However, since organic solvents are used in these inks, they have the problems, e.g., environmental pollution such as air pollution, labor safety and hygiene such as poisoning by organic solvent, and danger of igniting to cause explosion. As one means to solve the above problems, it has been proposed to use aqueous inks which utilize water and aqueous solvents. However, the aqueous inks suffer from the problem that they are inferior to conventional organic solvent type inks in drying property because water is contained in the ink compositions, when impermeable materials such as plastic film bases are printed with these aqueous inks.

[0005]

15 [Problem to be solved by the Invention]

Thus, the object of the present invention is to provide an aqueous ink composition which is less in danger, shows excellent drying property and is mild for earth environment.

20 [0006]

[Means for Solving Problem]

As a result of intensive research for attaining the above object, an aqueous ink composition containing a quick-drying property imparting agent which accelerates drying of ink has been found to accomplish the invention.

[0007]

The aqueous ink composition of the present invention is characterized by containing a quick-drying property imparting agent. By adding a quick-drying property imparting agent which is solid at room temperature and has a solubility in water lower than the solubility in the water-soluble solvent other than water, the ink drying property can be markedly improved.

[0008]

10 Since the quick-drying property imparting agent is low in solubility in water and solid at room temperature, it precipitates when a part of the water-soluble solvent other than water is evaporated, and can instantaneously fix dyes and others on the printing material to result in remarkable improvement of the drying property. Furthermore, it is also one factor for increase of drying property of ink that the quick-drying property imparting agent weakens the interaction between water molecules.

20 [0009]

The melting point of the quick-drying property imparting agent is preferably in the range of 20-250°C, more preferably 30-150°C. If the melting point of the quick-drying property imparting agent is lower than 25 20°C, the quick-drying property imparting agent is not precipitated at the time of drying, and this is not preferred. On the other hand, if the melting point of

the quick-drying property imparting agent is higher than 250°C, the effect is saturated and this is not preferred.

[0010]

5 Solubility of the quick-drying property imparting agent in water is preferably in the range of 0.1-60 wt%, more preferably 0.5-30 wt%. If it is less than 0.1 wt%, precipitates are apt to be produced when ink is kept at low temperatures. If it is more than 60  
10 wt%, the quick-drying property imparting agent does not precipitate at drying and this is not preferred.

[0011]

Solubility in the water-soluble solvent other than water is preferably in the range of 1-80 wt%, more  
15 preferably 5-50 wt%. If the solubility is less than 1 wt%, precipitates are apt to be produced at low temperatures. If it is more than 80 wt%, the quick-drying property imparting agent is sometimes not precipitated at the time of drying, and this is not  
20 preferred.

[0012]

Amount of the quick-drying property imparting agent in the aqueous ink composition of the present invention is preferably in the range of 1-10 wt%, more  
25 preferably in the range of 2-6 wt%. If the amount of the quick-drying property imparting agent is less than 1 wt%, the effect cannot sometimes be sufficiently

exhibited. On the other hand, if the amount of the quick-drying property imparting agent is more than 10 wt%, the effect is saturated and the addition is not much effective, and, besides, when the ink composition 5 is used as an ink for ink jet, sometimes this causes clogging of head and this is not preferred.

[0013]

The quick-drying property imparting agents added to the aqueous ink composition of the present 10 invention are preferably cyclic compounds having in the molecule at least one element of nitrogen, oxygen and sulfur elements and a water-soluble functional group. As the quick-drying property imparting agents, mention may be made of, for example, bemegride, benzalphthalide, 15 1,2,4-benzenetricarboxylic anhydride, benzil, benzimidazole, 2-benzimidazolepropionic acid, 2-benzimidazolylacetonitrile, benzo[c]cinnoline, benzo-12-crown-4, benzo-15-crown-5, benzo-18-crown-6, 1,4-benzodioxane-6-carboxyaldehyde, 3H-1,2-benzodithiol-3-one, 20 2-benzofurancarboxylic acid, benzofuroxane, 2,1,3-benzothiadiazole, 2H-1,4-benzothiazin-3(4H)-one, 1,2,3-benzotriazin-4(3H)-one, benzotriazole, benzotriazole-5-carboxylic acid, 1H-benzotriazole-1-methanol, 1-benzotriazolyl-9-fluorenylmethyl carbonate, N-(1H- 25 benzotriazol-1-ylmethyl)formamide, 2H-1,4-benzoxazin-3(4H)-one, benzoxazole, 2-benzoxazolinone, 2-benzoylthiophene, 2-benzylamino-4-methylpyridine, 4-

benzylamino-7-nitro-2,1,3-benzoxadiazole, 6-

benzylaminopurine, 2-benzylaminopyridine, (-)-2,3-O-

benzylidene-L-threitol, 1-benzylimidazole, N-

benzylmaleimide, (S)-(-)-4-benzyl-1-2-oxazolidinone, N-

5 (benzyloxycarbonyloxy) succinimide, 4-benzyloxy-2-(1H)-

pyridone, 4-benzyloxy-3-pyrrolin-2-one, 5-benzyl-1H-

pyrrolo[2,3-c]pyridine-3-carboxyaldehyde, N-

benzylphthalimide, 3,4-bis(acetoxymethyl)furan,

bis[(benzo-15-crown-5)-15-ylmethyl] pimelate, 1,4-bis(5-

10 phenyloxazol-2-yl)benzene, 1,2-bis(4-pyridyl)ethane,

1,2-bis(2-pyridyl)ethylene, 1,3-bis(3-pyridylmethyl)-2-

thiourea, 2,3-bis(2-pyridyl)pyrazine, N-(2-

bromobenzyloxycarbonyloxy) succinimide, butadiene

sulfone, 3-carboxy-1,4-dimethyl-2-pyrroleacetic acid, 2-

15 coumaranone, coumarin, coumarin-3-carboxylic acid, 18-

crown-6, dihydroascorbic acid, 3,4-dihydro-DL-proline,

3,5-diacetyl-1,4-dihydro-2,6-dimethylpyridine, 3,5-

diacetyl-2,6-dimethylpyridine, 1,3-diacetyl-2-

imidazolidinone, 2,6-diacetylpyridine, (+)-diacetyl-L-

20 tartaric anhydride, 3,5-diacetyltetrahydropyran-2,4,6-

trione, dibenzo-18-crown-6, dibenzo-24-crown-8,

dibenzo-30-crown-10, dibenzofuran, dibenzothiophene,

dibenzothiophene sulfone, 5,8-difluoro-1,4-benzodioxane,

diglycolic anhydride, dihydro-4,4-dimethyl-2,3-

25 furandione, 5,6-dihydro-5-methyl-4H-1,3,5-dithiazine,

2,5-dimercapto-1,3,4-thiadiazole, 4,4'-dimethyl-2,2'-

dipyridyl, dimethyl-3,4-furandicarboxylate, 2,3-

dimethylmaleic anhydride, ethylenediaminetetraacetic dianhydride, furfuryl sulfide, furil, homophthalic anhydride, 4-hydroxy-1,3-benzodioxol-2-one, 2-hydroxybenzothiazole, 6-hydroxy-1,3-benzoxathiol-2-one,  
5 N-(2-hydroxyethyl)phthalimide, N-hydroxysuccinimidyl acetoacetate, N-methylsuccinimide, N-phenylmaleimide, phthalazine, 1(2H)-phthalazione, phthalide, piperonal, piperonyl alcohol, piperonylic acid, 1-piperonylpiperazine, sesamol, 2-thiophenacetic acid, 3-  
10 thiophenemaleic acid, and the like. Among them, preferred are compounds having a functional group and an aromatic ring in the molecule, such as 2-mercaptopbenzoxazole, benzotriazole, 2-benzooxazolinone, and phthalimide. Benzotriazole compounds are particularly  
15 preferred because they have rust proofing action and can inhibit formation of rusts in the devices used, such as printers.

[0014]

As the water-soluble solvents added to the aqueous ink composition of the present invention, mention may be made of, for example, alcohols such as methyl alcohol, ethyl alcohol, n-butyl alcohol, isobutyl alcohol, tert-butyl alcohol, n-propyl alcohol and isopropyl alcohol; amides such as dimethyl-formaldehyde and dimethylacetamide; ketones such as acetone and methyl ethyl ketone; ethers such as tetrahydrofuran, dioxane, ethylene glycol methyl ether, ethylene glycol  
25

ethyl ether, diethylene glycol methyl ether, diethylene glycol ethyl ether, triethylene glycol monomethyl ether and triethylene glycol monoethyl ether; polyhydric alcohols such as ethylene glycol, propylene glycol,  
5 butylene glycol, triethylene glycol, 1,2,6-hexanetriol, thioglycol, diethylene glycol, polyethylene glycol, polypropylene glycol and glycerin; N-methyl-pyrrolidone, 1,3-dimethyl-2-imidazolidinone, and the like. These may be used in combination.

10 [0015]

As the fluorescent dyes, preferred are those which comprise a rare earth element and a ligand. The rare earth element which is a luminescence center of the fluorescent dyes preferably comprises at least one  
15 element selected from europium, dysprosium, terbium, neodymium, praseodymium, samarium, gadolinium, holmium, erbium, and thulium. These elements can form stable fluorescent complexes and a sufficient emission intensity can be obtained.

20 [0016]

Among the above rare earth elements, europium is most preferred in the case of application of the aqueous ink composition to security, FA, various cards, bar code systems or the like. When europium is the  
25 luminescence center, the emission has red color and  $615 \pm 20$  nm. Therefore, printed marks emit visible light on the above longer wavelength side by excitation with

ultraviolet rays, and, hence, the color of background gives little influence and detection can be performed with high sensitivity by a silicon photo diode or the like. The emission of blue or green is sometimes difficult to detect. When a mark which emits blue light is formed on a white paper impregnated with a fluorescent brightener, since the background also emits light, the difference in light volume of emission substantially decreases, and the detection is sometimes impossible. Furthermore, if a silicon photo diode which is generally inexpensive and easily available is used as an optoelectric transducer in detection of emitted visible light, photoreceiving sensitivity for the visible light becomes lower on the shorter wavelength side than the longer wavelength side, and the sensitivity for blue or green visible light of relatively short wavelength is less than half the sensitivity for the visible light of longer wavelength side of about 600 nm, and, thus, sufficient detection sensitivity cannot sometimes be obtained.

[0017]

The ligands include, for example, thenoyltrifluoroacetone, naphthoyltrifluoroacetone, benzoyltrifluoroacetone, methylbenzoyltrifluoroacetone, furoyltrifluoroacetone, pivaloyltrifluoroacetone, hexafluoroacetylacetone, trifluoroacetylacetone, fluoroacetylacetone, heptafluorobutanoylpivaloylmethane,

8-hydroquinoline, 8-mercaptoquinoline, tri-n-butyl phosphate, tri-n-butylphosphine oxide, tri-n-octylphosphine oxide, di-n-butyl sulfoxide, pyridine,  $\alpha$ -picoline,  $\beta$ -picoline,  $\gamma$ -picoline, piperidine, 5 quinoline, and the like. Among them, thenoyltrifluoroacetone and naphthoyltrifluoroacetone are especially preferred.

[0018]

The fluorescent dyes can be produced by 10 suitable methods known to one skilled in the art. For example, it can be easily obtained by reacting a ligand such as acetylacetone with a rare earth metal halide such as europium chloride under proper conditions.

[0019]

15 It is preferred that the aqueous ink composition of the present invention contains water in an amount of 30-95 wt%. More preferred content of water in the aqueous ink composition is 40-80 wt%. By containing not less than 30 wt% of water, there can be 20 provided aqueous ink compositions harmless for human bodies and excellent in safety.

[0020]

The aqueous ink composition of the present invention can be prepared using medium stirring mills, 25 for example, container driving medium mills such as ball mills, centrifugal mills and planetary ball mills, high-speed rotary mills such as sand mills, and stirring tank

type mills, or simple dispersing machines such as disper.

[0021]

The aqueous ink composition of the present invention may contain various additives which are contained in usual ink compositions, such as binders, charge donating agents, pH adjusters, fluorescent sensitizers, surface active agents, leveling agents, anti-foaming agents, germicides, and antioxidants. The binders include, for example, vinyl resins such as polyvinyl alcohol, polyvinyl butyral and polyvinyl acetate, urethane resins, phenolic resins, polyester resins, acrylic resins, cellulosic resins, polyamides, maleic resins, and copolymers thereof. The charge regulators include, for example, lithium salts such as LiNO<sub>3</sub>, potassium salts such as KCN and KSCN, and cation compounds such as tetraphenylphosphonium bromide. The pH adjusters include, for example, amine compounds such as diethanolamine, triethanolamine and triethylene-tetramine, amide compounds, hydroxides such as lithium hydroxide, sodium hydroxide and potassium hydroxide, and carbonates. The fluorescent sensitizers include, for example, phosphorus compounds such as phosphine oxide compounds, phosphine sulfide compounds and phosphine compounds, and nitrogen-containing organic compounds such as benzotriazole.

[0022]

Content of the fluorescent dye in the ink composition is preferably 0.1-10 wt%, more preferably 0.5-5 wt%. When the content is 0.1 wt% or more, 5 emission intensity may not be reduced, and when it is 10 wt% or less, concentration quenching can be prevented and emission intensity can be prevented from additional reduction.

[0023]

10 The prints produced using the aqueous ink composition of the present invention are completely or substantially invisible to the naked eyes and cannot be identified. The prints produced using a fluorescent dye containing a rare earth element such as europium can be 15 recognized only when they are irradiated with ultra-violet rays and emit red light in the visible light region. When neodymium is used as the rare earth element of the luminescence center, the prints emit light in the infrared region upon excitation with 20 infrared rays and can be detected only by an exclusive detector. These prints of the present invention which emit visible light and infrared light are both normally invisible, and due to these features, the ink composition of the present invention can be applied to 25 various cards such as for security, FA and others.

[0024]

When bar codes are printed with the aqueous

ink compositions of the present invention, black and white bar code printing can be improved. That is, the conventional black and white bar code printing has the defect that it damages the appearance of articles while

5 according to the ink composition of the present invention this defect can be overcome. The bar code printing which uses the aqueous ink composition of the present invention can also be utilized for mailing to which a system of distributing postal matters classified

10 by code control.

[0025]

As methods for making the above prints, there may be employed all of the known printing methods, such as ink jet printing, offset printing, gravure printing, heat sensitive transfer printing, and the like. The aqueous ink compositions of the present invention are especially suitable for ink jet printing. Since the ink compositions of the present invention are dissolved or stably dispersed in aqueous solvents, they do not cause

15 clogging of nozzles of ink jet printers and can be stably discharged from the nozzles.

20

[0026]

Furthermore, the aqueous ink compositions of the present invention may be used as inks for all systems such as ink jet printing, offset printing, gravure printing, heat sensitive transfer printing, and the like, but ink jet printing is more preferred.

[0027]

[Mode for Carrying Out the Invention]

[Embodiments]

The aqueous ink composition of the present  
5 invention will be specifically illustrated. All parts  
are by weight.

[0028]

Example 1

	Thenoyltrifluoroacetone	12.6 parts
10	Ethanol	274.0 parts
	Europium (III) chloride · hexahydrate	3.0 parts
	Deionized water	274.0 parts

6.9 Parts of dimethylethanolamine was added dropwise to the above components under being stirred by 15 a magnetic stirrer to adjust the pH to 6.8. Then, thereto was added 37.2 parts of polyvinyl pyrrolidone K25 (manufactured by Wako Jun-Yaku Kogyo Co., Ltd., average molecular weight 25000), followed by stirring at 60°C for 3 hours and then filtration to obtain a 20 phosphor solution A.

[0029]

Furthermore, to the resulting phosphor solution A were added 0.2 part of BYK-348 (manufactured by BYK Chemie Co., Ltd.) as a leveling agent and 17.0 25 parts of 1,2,3-benzotriazole as a quick-drying property imparting agent, followed by stirring at 25°C for 1 hour

and then filtration to obtain an aqueous ink composition

A.

[0030]

Example 2

5 An aqueous ink composition B was obtained in  
the same manner as in Example 1, except that 1,2,3-  
benzotriazole of the quick-drying property imparting  
agent used in Example 1 was changed to benzoxazole.

[0031]

10 Example 3

An aqueous ink composition C was obtained in  
the same manner as in Example 1, except that 1,2,3-  
benzotriazole of the quick-drying property imparting  
agent used in Example 1 was changed to 2-  
15 benzoxazolinone.

[0032]

Example 4

An aqueous ink composition D was obtained in  
the same manner as in Example 1, except that neodymium  
20 chloride · hexahydrate was used in place of europium  
chloride · hexahydrate used in Example 1.

[0033]

Comparative Example 1

	Thenoyltrifluoroacetone	12.6 parts
25	Ethanol	274.0 parts
	Europium (III) chloride · hexahydrate	3.0 parts
	Deionized water	274.0 parts

6.9 Parts of dimethylethanolamine was added dropwise to the above components under being stirred by a magnetic stirrer to adjust the pH to 6.8. Then, thereto was added 37.2 parts of polyvinyl pyrrolidone K25 (manufactured by Wako Jun-Yaku Kogyo Co., Ltd., average molecular weight 25000), followed by stirring at 60°C for 3 hours and then filtration to obtain a phosphor solution E.

[0034]

Furthermore, to the resulting phosphor solution E was added 0.2 part of BYK-348 (manufactured by BYK Chemie Co., Ltd.) as a leveling agent, followed by stirring at 25°C for 1 hour and then filtration to obtain an aqueous ink composition E.

[0035]

Comparative Example 2

Thenoyltrifluoroacetone	12.6 parts
Ethanol	548.0 parts
Europium (III) chloride · hexahydrate	3.0 parts

6.9 Parts of dimethylethanolamine was added dropwise to the above components under being stirred by a magnetic stirrer to adjust the pH to 6.8. Then, thereto was added 37.2 parts of polyvinyl pyrrolidone K25 (manufactured by Wako Jun-Yaku Kogyo Co., Ltd., average molecular weight 25000), followed by stirring at 60°C for 3 hours and then filtration to obtain a phosphor solution F.

[0036]

Furthermore, to the resulting phosphor solution F was added 0.2 part of BYK-348 (manufactured by BYK Chemie Co., Ltd.) as a leveling agent, and 17.0 5 parts of 1,2,3-benzotriazole as a quick-drying property imparting agent, followed by stirring at 25°C for 1 hour and then filtration to obtain an aqueous ink composition F.

[0037]

10 (Drying time of Print)

Printing was carried out on PET films by an ink jet printer CB3 manufactured by Domino Co., Ltd. After lapse of a given time, the print was rubbed with a brush for oil paint and the time before the print disappeared 15 by the rubbing was measured.

[0038]

(Emission intensity of ink and print)

The emission intensity of ink and print was measured by a spectrophotofluorometer (FP750 20 manufactured by Nihon Bunko Co., Ltd.). As for the emission intensity, evaluation was made on prints produced by carrying out printing on plain papers (manufactured by XEROX Co.) with the ink compositions by an ink jet printer CB3 manufactured by Domino Co., Ltd. 25 Relative comparison was made on the emission intensity with assuming the ethanol based ink composition containing no water in the solvent of Comparative

Example 2 to be 100. The prints other than that produced using the ink composition L were irradiated with exciting light of 365 nm and the emission peak intensity at 615 nm was used for comparison, and the  
5 print produced using the ink composition L was irradiated with an exciting light of 810 nm and the emission peak intensity at 1065 nm was used for comparison. Results of evaluation in Examples 1-4 and Comparative Examples 1-2 are shown in Table 1.

[0039]

[Table 1]

	Ink composition	Ink solvent	Drying time of print (sec)	Emission intensity of ink	Emission intensity of print
Example 1	Ink composition A	Ethanol/water	0.6	110	110
Example 2	Ink composition B	Ethanol/water	0.6	100	120
Example 3	Ink composition C	Ethanol/water	0.6	90	120
Example 4	Ink composition D	Ethanol/water	0.6	100	100
Comparative Example 1	Ink composition E	Ethanol/water	5	100	120
Comparative Example 2	Ink composition F	Ethanol	0.6	100	100

[0040]

From the results shown in Table 1, it was confirmed that the ink compositions A-D of the present invention had drying property better than that of the conventional ink compositions E to which the quick-drying property imparting agent was not added. Moreover, it was confirmed that the ink compositions of the present invention had drying property similar to that of the organic solvent type ink composition D containing no water.

[0041]

Furthermore, it was confirmed that the emission intensity of the ink and print was equal to or higher than that in the comparative examples.

15 [0042]

[Effects of the Invention]

As explained above, the aqueous ink compositions of the present invention can show excellent drying property as compared with the conventional aqueous ink compositions. Furthermore, there can be obtained safe aqueous ink compositions which are mild for environment and less in problems, namely, environmental pollution such as air pollution, labor safety and hygiene such as organic solvent poisoning, and danger of ignition and explosion.

[Title of Document] Abstract

[Abstract]

[Problem] To provide an aqueous ink composition which shows excellent drying characteristics and which is less in danger and mild for earth environment.

[Solving Means] The aqueous ink composition of the present invention contains a quick-drying property imparting agent which accelerates drying of ink.

[Selected Drawing] No.